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*Indian Standard*

METHODS OF SAMPLING AND  
TEST FOR DRYING OILS FOR PAINTS

( *Second Revision* )

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BUREAU OF INDIAN STANDARDS  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

# *Indian Standard*

## METHODS OF SAMPLING AND TEST FOR DRYING OILS FOR PAINTS

### ( *Second Revision* )

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# *Indian Standard*

## METHODS OF SAMPLING AND TEST FOR DRYING OILS FOR PAINTS

### *( Second Revision )*

#### 0. FOREWORD

**0.1** This Indian Standard ( Second Revision ) was adopted by the Indian Standards Institution on 17 August 1979, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was first published in 1950 and subsequently revised in 1966. In this second revision additional methods of test for colour-change on heating, freedom from fish oil, linseed oil, blown oil and cottonseed oil have been included. For testing presence of castor oil in other oils, two methods, namely ammonium molybdate test and thin layer chromatographic procedure, in line with those prescribed in IS : 548 ( Part II )-1976\*, have been included. A method for determination of rosin acids has also been included. The term 'specific gravity' has been given as 'relative density' according to the latest international practice.

**0.3** This standard contains clauses 3.3.4, 3.4.1.2 and 3.4.2.4 which call for agreements between the purchaser and the supplier.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1970†.

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#### 1. SCOPE

**1.1** This standard prescribes methods of sampling and test for drying oils for paints.

#### 2. TERMINOLOGY

**2.1** For the purpose of this standard, definitions given in IS : 1303-1963‡ and the following shall apply.

\*Methods of sampling and test for oils and fats : Part II Purity tests ( *third revision* ).

†Rules for rounding off numerical values ( *revised* ).

‡Glossary of terms relating to paints ( *revised* ).



**2.1.1 Approved Sample** — The sample accepted by the indenter or inspection authority as the basis for supply. When a sample is tested and approved by the purchaser or inspection authority, the result of such tests as permits the supplier to meet the limits imposed by the specification for deliveries shall be made available to the supplier.

**2.1.2 Freedom from Lead (Lead Free)** — It shall mean that the material does not contain lead or compounds of lead or a mixture of both, calculated as metallic lead (Pb), exceeding 0.03 percent by mass.

**2.1.3 Room Temperature** — It shall mean a temperature between 21 and 38°C unless otherwise specified.

**2.1.4 Standard Atmospheric Conditions for Testing** — An atmosphere with a relative humidity of  $65 \pm 2$  percent and a temperature of  $27 \pm 2^\circ\text{C}$  provided that in a given series of tests, the temperature does not vary by more than  $\pm 1^\circ\text{C}$  (see also IS : 196-1966\*).

**2.1.5 Surface Dry** — A condition of the oil film such that when clean, dry, silver sand, graded so as to pass 300-micron IS Sieve (see IS : 460-1962†) but to be retained on 150-micron IS Sieve, sprinkled on to the surface of the film and allowed to remain for one minute, could be removed, without injury to the paint film, by means of a camel hair brush.

**2.1.6 Tender Sample** — The sample submitted by the supplier with his tender.

### 3. SAMPLING

**3.1 General** — In drawing samples, the following precautions and directions shall be observed.

**3.1.1** Samples shall not be taken in an exposed place.

**3.1.2** The sampling apparatus shall be clean and dry.

**3.1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling apparatus and the containers for samples from adventitious contamination.

**3.1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by shaking, stirring, or both, by suitable means, or by rolling.

**3.1.5** The samples shall be placed in clean, dry and airtight metal or opaque glass containers on which the material has no action.

\*Atmospheric conditions for testing (revised).

†Specification for test sieves (revised).

3.1.6 The sample containers shall be of such a size that they are almost completely filled with the sample.

3.1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling and the month and year of manufacture of the material.

3.1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

## 3.2 Sampling Instruments

3.2.1 *Sampling Bottle or Can* (see Fig. 1) — This instrument is suitable for sampling large vessels and tanks of liquid oil. It consists of a weighted bottle or metal container with removable stopper or of a cork, to which is attached a suitable chain or cord. The bottle or can is fastened to a pole. It is lowered to the required depth in the tank and the stopper is removed by means of chain or cord for filling the container.

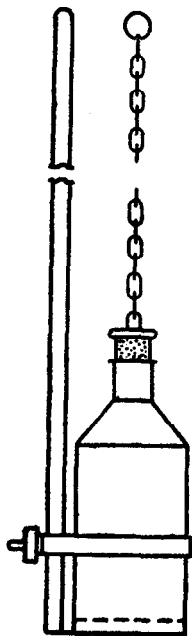


FIG 1. SAMPLING BOTTLE OR CAN

**3.2.2 Sampling Tubes** — The recommended forms of sampling tubes are:

- a) Closed-type sampling tube, undivided or divided, for sampling liquids that may not be homogeneous; and
- b) Open type sampling tube, for sampling homogeneous liquids.

**3.2.2.1 Closed-type sampling tube, undivided** (see Fig. 2) — It consists of two concentric metallic tubes closely fitted into each other throughout their entire length, so that one tube may be rotated within the other. Longitudinal openings of about one-third of the circumference are cut in both tubes. In one position the openings in the two tubes coincide; the sampling tube is open when in this position and admits the material. By turning the inner tube through an angle of  $180^\circ$ , it becomes a sealed container. The inner tube which has a diameter of 20 to 40 mm is undivided along its length to serve as a single container. The two concentric tubes are provided with ports at their bottoms. The material shall be drained out from the container through these ports when the longitudinal openings in the two tubes coincide. The length of the instrument shall be such as to enable it to reach the bottom of the container being sampled. The instrument is inserted closed, the material is admitted by opening it, and finally, it is closed and withdrawn.

**3.2.2.2 Close-type sampling tube, divided** (see Fig. 3) — It consists of metal and has D-shaped cross section. It is provided with compartments along its length and is opened and closed by means of a closely fitting shutter which moves up and down throughout the entire length. It shall be from 25 to 50 mm wide. The instrument is inserted closed, the shutter is pulled out to admit the material, and the tube is then closed and withdrawn.

**3.2.2.3 Open-type sampling tube for homogeneous liquids** (see Fig. 4) — It is made of metal or thick glass, and shall be of 20 to 40 mm diameter and 400 to 800 mm length. The upper and lower ends are conical and narrow down to 5 to 10 mm diameter. Handling is facilitated by two rings at the upper end. For taking a sample, the instrument is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

### 3.3 Scale of Sampling

**3.3.1 Lot** — In a single consignment, all containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each group shall constitute a separate lot. In case the consignment is in tanks, the tanks belonging to the same batch of manufacture shall constitute a lot.

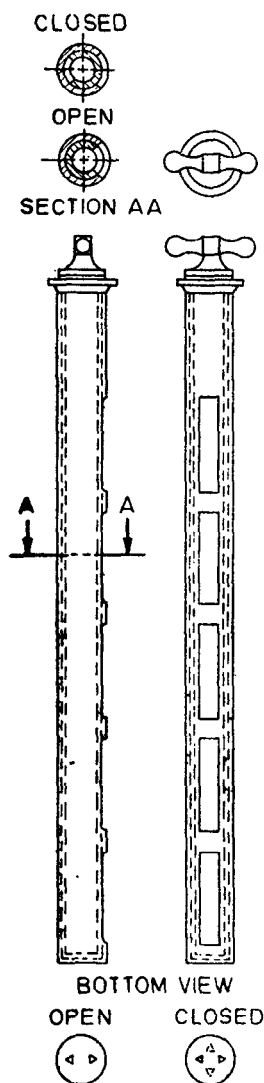


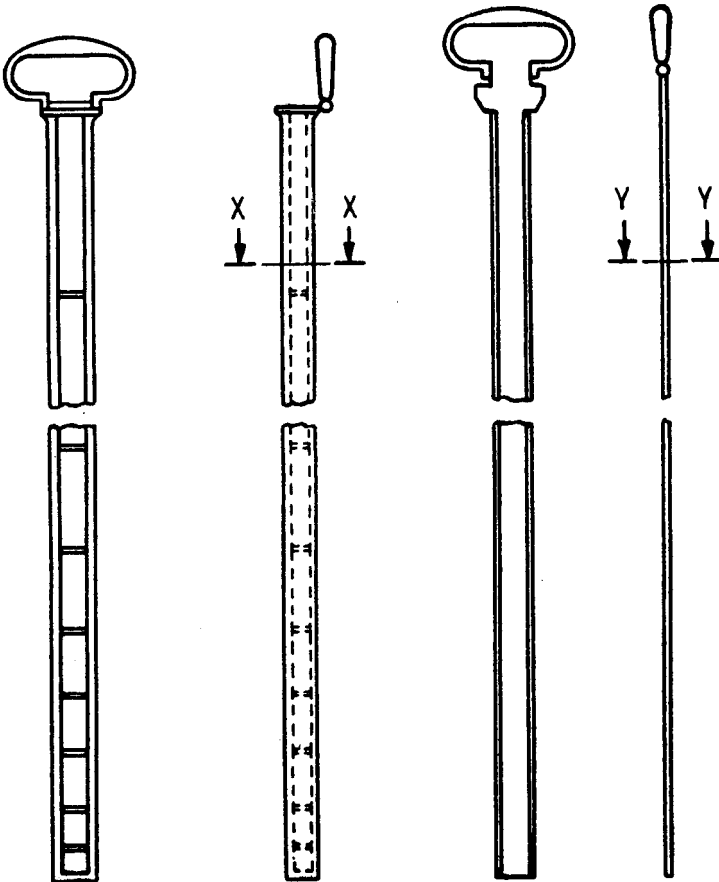
FIG. 2 CLOSED-TYPE SAMPLING TUBE, UNDIVIDED



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SECTION XX



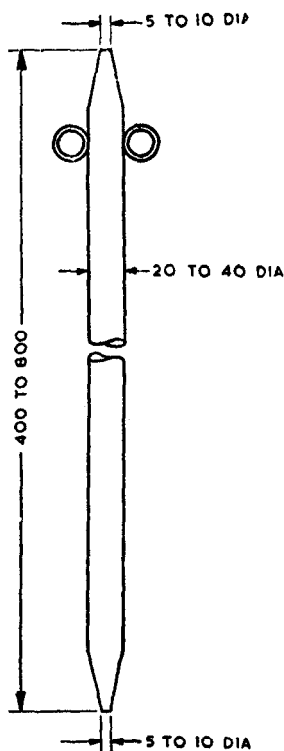
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SECTION YY



TUBE

SHUTTER

FIG. 3 CLOSED-TYPE SAMPLING TUBE, DIVIDED



All dimensions in millimetres

FIG. 4 OPEN-TYPE SAMPLING TUBE FOR HOMOGENEOUS LIQUIDS

**3.3.1.1** For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately.

**3.3.2 Sampling from Tanks**— Each tank in the lot shall be sampled separately for determining the conformity of the lot to the requirements of the specification.

**3.3.3 Sampling from Containers**— The number of containers ( $n$ ) to be selected from the lot shall depend upon the size of the lot ( $N$ ) and shall be in accordance with Table 1.

**3.3.4** These containers shall be selected at random from the lot. To ensure the randomness of selection, a random number table, as agreed

between the purchaser and the supplier, shall be used. In case such a table is not available, the following procedure shall be adopted :

Starting from any container, count them as 1, 2, 3.....up to  $r$  and so on, in one order, where  $r$  is the integral part of  $N/n$ . Every  $r$ th container thus counted shall be withdrawn to give the sample for test.

**TABLE 1 SCALE OF SAMPLING FOR DRYING OILS FOR PAINTS**

( Clause 3.3.3 )

VOLUME OF SUPPLY IN A LOT ( $N$ )	No. of CONTAINERS TO BE SAMPLED FOR SIZE OF CONTAINERS ( $n$ )				
	Above 20 Litre	20 Litre	10 Litre	4 or 5 Litre	2 Litre or Less
Litres					
Up to 50	1	1	1	1	1
51 „ 100	1	1	1	1	3
101 „ 150	1	1	1	2	3
151 „ 200	1	2	2	2	3
201 „ 300	1	2	2	2	4
301 „ 400	1	2	2	3	5
401 „ 500	1	2	3	3	5
501 „ 600	1	2	3	3	5
601 „ 750	1	2	3	3	6
751 „ 1 000	2	3	3	4	6
1 001 „ 1 500	2	3	3	4	7
1 501 „ 2 000	2	3	4	5	8
2 001 „ 2 500	2	3	4	5	9
2 501 „ 3 000	2	4	4	5	9
3 001 „ 3 500	2	4	5	5	10
3 501 „ 4 250	3	4	5	6	10
4 251 „ 5 000	3	4	5	6	11
5 001 „ 6 000	3	5	6	7	12
6 001 „ 7 500	3	5	6	7	13
7 501 „ 10 000	3	5	6	8	14
10 001 „ 15 000	4	6	7	9	15
15 001 „ 20 000	4	7	8	10	17
20 001 „ 25 000	5	7	9	11	19
25 001 „ 30 000	5	8	9	11	20
30 001 „ 35 000	5	8	10	12	21
35 001 „ 42 500	6	9	11	13	22
42 501 „ 50 000	6	9	11	13	24

### 3.4 Procedure for Drawing Samples

**3.4.1 Test Samples from Tanks** — The sample shall be taken before transferring the contents of the tank into smaller containers and also once on every eighth day as long as there remains some material in the tank. For drawing the sample from the tank, lower the closed sampling bottle or can slowly to the required depth, open and fill it at that depth. If the material is suspected to be non-homogeneous, three samples shall be obtained at levels of one-tenth of the depth of the liquid from the surface (top sample), one-half of the depth (middle sample), and nine-tenth of the depth of the liquid from the top surface (lower sample). If foots, or water, or both are present, a fourth sample shall be taken at the lowest point of the container (bottom sample). All the three/four samples, thus obtained from a tank, shall be subjected to preliminary examination (see 3.5), record observations and if the samples are free from foreign matter and visible impurities, they shall then be mixed in a clean dry container. This sample shall be further divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee. Each tank in the lot shall be sampled in the above manner and separate samples shall be obtained from each of the tanks. The approximate quantity of the material to be drawn from a tank shall nearly be equal to thrice the quantity required for carrying out tests for the requirements prescribed in this standard.

**3.4.1.1** All the samples thus obtained from tanks in the lot shall be transferred to separate containers. These containers shall then be sealed airtight with stoppers and labelled with full identification particulars given in 3.1.7.

**3.4.1.2** The referee test samples, consisting of the samples from the tanks in the lot, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in the case of any dispute.

**3.4.2 Test Samples from Containers** — To ensure that the samples taken from each container are fairly representative, the contents shall be mixed thoroughly by shaking or stirring or rolling. Draw small samples of the material from various depths with the help of a suitable sampling tube depending on whether the material is homogeneous or not. The approximate quantity of the material to be drawn from a container shall nearly be equal to thrice the quantity required for testing purposes but in no case shall be less than 500 ml.

**3.4.2.1** Out of the material drawn from individual containers, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determination of all characteristics. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.



**3.4.2.2** The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the  $n$  containers shall be for the purchaser, another for the supplier and the third for the referee.

**3.4.2.3** All the individual and composite samples shall be transferred to separate sample containers. These containers shall then be sealed airtight with stoppers and labelled with full identification particulars given in 3.1.7.

**3.4.2.4** The referee test samples, consisting of a composite sample and a set of  $n$  individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in case of any dispute.

**3.5 Preliminary Examination of Samples** — At the time of drawing samples either from tanks or containers, each sample shall be tested for the presence of foreign matter and visible impurities soon after sampling and observations shall be recorded.

### 3.6 Number of Tests

**3.6.1 For Samples from Tanks** — Tests for determination of all characteristics in the specification shall be conducted on the sample obtained from each tank.

**3.6.2 For Samples from Containers** — Tests for determination of all characteristics shall be conducted on the composite sample. However, in case of doubt regarding the non-uniformity of the material in a particular batch or lot, or if the purchaser desires, tests shall be conducted on the individual samples.

### 3.7 Criteria for Conformity

**3.7.1 For Tanks** — The lot shall be considered as conforming to the requirements of the relevant standard if the test sample from each tank satisfies all the requirements specified in that standard.

**3.7.2 For Containers** — The lot shall be considered as conforming to the requirements of the relevant standard if the different test results obtained on individual or composite sample, as the case may be, satisfy all the requirements specified in that standard.

## 4. QUALITY OF REAGENTS

**4.1** Unless otherwise specified, pure chemicals and distilled water (see IS : 1070-1977\*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

\*Specification for water for general laboratory use (second revision).

## 5. COLOUR

**5.1** The colour shall be determined in an all-glass cell of specified dimensions in a standard Tintometer ( Lovibond type is recommended ) or by comparison in a colorimeter with the specified standard.

## 6. RELATIVE DENSITY

**6.1 Outline of the Method** — The relative density may be determined with a standard hydrometer or Westphal hydrostatic balance or with a relative density bottle or pycnometer. The relative density bottle or pycnometer method shall be adopted as referee method in case of dispute. The temperature at which the relative density is determined shall be reported.

**6.1 Temperature Corrections** — In certain cases, it may be inconvenient to determine the relative density at the specified temperature, in which case, it may be determined at a convenient temperature and the results corrected as given in 6.1.1 and 6.1.2.

**6.1.1** If the temperature of testing is higher than the specified temperature, add 0.000 65 to the value of the relative density obtained for each degree celsius of the difference between the two temperature.

**6.1.2** If the temperature of testing is lower than the specified temperature, subtract 0.000 65 for each degree celsius of the difference between the two temperatures.

## 6.2 Procedure

**6.2.1 Hydrometer Method** — Pour the material to be tested into a clean hydrometer jar without splashing, so as to avoid the formation of air bubbles. The diameter of the jar shall be at least 2.5 cm greater than the diameter of the hydrometer to be used therein and the hydrometer shall not touch the sides of the jar. If air bubbles are formed, remove them after they have collected on the surface by touching them with a piece of clean blotting paper or filter paper. Keep the jar in a vertical position in a place free from air currents and in a bath maintained at the specified temperature. Lower the hydrometer gently into the sample, and when it has settled, depress it about two scale divisions into the liquid, the unimmersed portion of the stem being kept dry, as any unnecessary liquid on the stem changes the effective mass of the instrument and affects the reading obtained. Allow sufficient time for the hydrometer to become completely stationary and for all air bubbles to come to the surface. Read the point on the hydrometer scale to which the material rises with the eye placed at the surface of the liquid.

**NOTE** — The use of the hydrometer method is not recommended for oils of high viscosity.

**6.2.2 Westphal Hydrostatic Balance Method** — Suspend the plummet in the cylinder filled with recently boiled water at the specified temperature and place the largest rider on the hook. The plummet shall not touch the sides of the cylinder. Adjust the screw on the base until the pointer is exactly opposite the fixed indicator point. Wipe the plummet and the cylinder to remove the water. Fill the cylinder with the material at the same temperature and dip the plummet into the liquid removing air bubbles, if any, formed in the eyehole of the plummet by lifting it from the liquid. Re-immerses the plummet and adjust the height of the balance till the plummet is nearly in the middle of the column of the liquid. Place riders on the beam till the pointer and the fixed indicator are exactly opposite each other. Read the relative density from the position of the riders on the beam, beginning with the largest and ending with the smallest.

### **6.2.3 Relative Density Bottle or Pyknometer Method**

#### **6.2.3.1 Apparatus**

- a) *Relative density bottle or pyknometer* — with well-fitting ground-glass joints. To calibrate, clean and dry the bottle or pyknometer thoroughly, weigh and then fill with recently boiled and cooled water at about 25°C after removing the cap of the side arm. Fill to overflowing by holding the bottle or pyknometer on its side in such a manner as to prevent the entrapment of air bubbles. Insert the stopper and immerse in a water-bath at the desired test temperature  $\pm 0.2^\circ\text{C}$ . Keep the entire bulb covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, replace the cap, cool to room temperature and weigh. Calculate the mass of water. This is a constant for the bottle or pyknometer, but should be checked periodically. A relative density bottle of about 50 ml capacity and of a either of the two shapes as shown in Fig. 5 is recommended.
- b) *Water-bath* — maintained at  $30.0 \pm 0.2^\circ\text{C}$ .
- c) *Thermometer* — any convenient thermometer of a suitable range with 0.1 or 0.2°C subdivisions.

**6.2.3.2 Procedure** — Fill the bottle with the oil previously cooled to about 25°C, holding the bottle on its side in such a manner as to prevent the entrapment of air bubbles after removing the cap of the side arm. Insert the stopper, immerse in the water-bath at  $30.0 \pm 0.2^\circ\text{C}$  and hold for 30 minutes. Carefully wipe off any oil which has come through the capillary opening. Remove the bottle from the bath, clean and dry it thoroughly. Replace the cap of the side arm, cool to room temperature and weigh.

#### **6.2.3.3 Calculation**

$$\text{Relative density at } 30^\circ\text{C}/30^\circ\text{C} = \frac{A - B}{C - B}$$

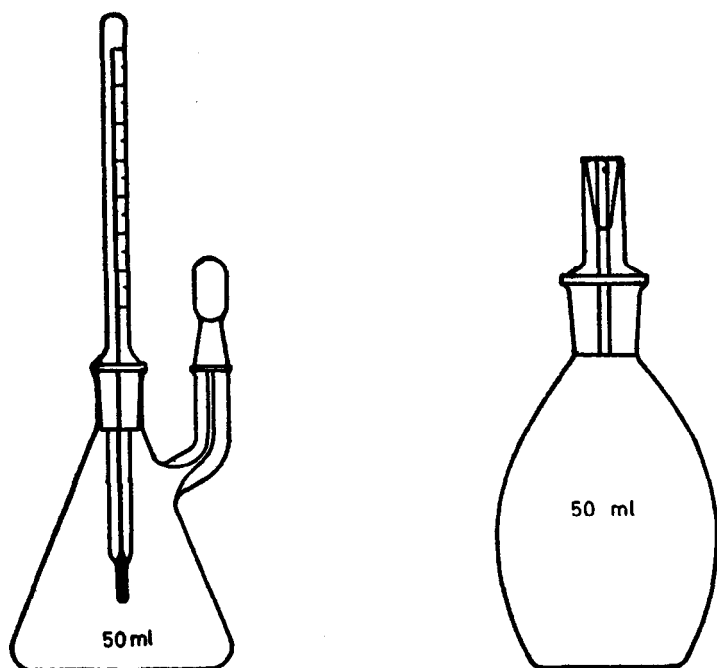


FIG. 5 RELATIVE DENSITY BOTTLES

where

$A$  = mass in g of the relative density bottle with oil at 30°C,

$B$  = mass in g of the relative density bottle, and

$C$  = mass in g of the relative density bottle with water at 30°C.

**6.2.3.4 Temperature corrections** — If the relative density is determined at any other temperature, then temperature corrections as given under 6.1 shall be applied.

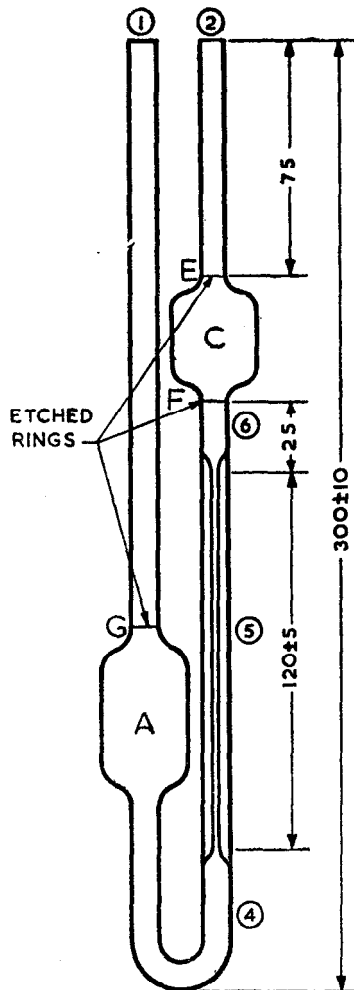
## 7. VISCOSITY

**7.1 Outline of the Method** — Viscosity of the material is determined with the Ostwald U-tube viscometer.

### 7.1 Apparatus

**7.1.1 U-tube Viscometer** — The U-tube viscometer shall be of homogeneous transparent glass and free from mechanical imperfections. All glass tubing employed in the construction of the viscometer shall be of the

same composition and the finished instrument shall be thoroughly annealed. The design, size and the dimensions of the viscometer to be used for various viscosity ranges are shown in Fig. 6 and Table 2.



All dimensions in millimetres

FIG. 6 OSTWALD U-TUBE VISCOMETER

**7.1.2 Thermometer** — Any convenient thermometer of suitable range with 0.1° to 0.2°C subdivisions.

**TABLE 2 DIMENSIONS OF U-TUBE VISCOMETERS***( Clause 7.1.1 and Fig. 6 )*

i) Size	B	C	D
ii) Viscosity range, cSt	4 to 10	7.5 to 35	20 to 120
iii) Tubes 1 and 4, mm	8 to 9 OD and	1 to 1.25 wall	9 to 10 OD and 1 to 1.25 wall
iv) Bulb A:			
a) Made from tubing mm	21 to 23 OD and	1 to 1.5 wall	25 to 27 OD and 1 to 1.5 wall
b) Length of parallel sides, Min, mm	20	20	28
v) Bore of capillary 5, ± 0.01 mm	0.71	0.88	1.4
vi) Tubes 2 and 6, mm	6 to 7 OD and	1 to 1.25w all	7 to 8 OD and 1 to 1.25 wall
vii) Bulb C:			
a) Made from tubing, mm	21 to 23 OD and	1 to 1.5 wall	25 to 27 OD and 1 to 1.5 wall
b) Capacity, ml	5.0 ± 0.25	5.0 ± 0.25	10.0 ± 0.5
viii) Vertical distance FG, mm	87 ± 4	83 ± 4	78 ± 4
ix) Distance between cen- tres of tubes 1 and 2, mm	20	20	20

**7.1.3 Bath** — A water-bath, suitable for immersion of viscometer within 5 cm of the top with provisions for visibility of the instrument and the thermometer, shall be provided. The viscometer may be fixed as an integral part of the bath. The thermometer shall be mounted with the bulb near the centre of the viscometer. The bath shall be regulated so that the variation in the thermometer reading does not exceed 0.02°C. Use of thermo-regulator is recommended.

**7.1.4 Time-Recording Device** — A suitable stop-watch.

**7.1.5 Frame** — Suitable frame shall be provided so that the viscometer may be suspended securely in the bath in a vertical position.

## 7.2 Procedure

**7.2.1** Select an appropriate U-tube viscometer and clean it by rinsing with suitable solvents, such as benzene (*see* IS : 534-1965\*) followed by ethyl ether (*see* IS : 336-1973†) or petroleum hydrocarbon solvents (*see* IS : 1745-1978‡). Remove each solvent by passing a current of dry air through it and

\*Specification for benzene (*revised*).

†Specification for ether (*second revision*).

‡Specification for petroleum hydrocarbon solvents (*second revision*).

take care that no moisture remains inside the instrument. Periodically or wherever necessary, clean the instrument by use of chromic acid cleaning solution, allowing it to stand in the instrument for several hours. After cleaning, rinse the viscometer with water, clean and dry with the solvents as given above.

**7.2.2** Suspend the cleaned viscometer in the bath maintained at  $30 \pm 0.02^\circ\text{C}$ , taking care to ensure that the capillary arm is vertical. Fill the viscometer through tube 1 with the filtered material, using a long pipette to minimize any wetting of the tube above the filling mark *G*. Allow at least 20 minutes for the viscometer to reach the test temperature and adjust the volume to bring the liquid within 0.2 mm of the filling mark. After the sample has reached the test temperature, apply suction or pressure to bring the liquid level up to a point 1 cm above the timing mark *E*. Release the suction or pressure and measure the time required from the bottom of the meniscus to pass from the top edge of mark *E* to the top edge of mark *F*.

### 7.3 Calculation

$$V = Ct - \frac{B}{t}$$

where

*V* = kinematic viscosity in centistokes,

*C* = constant for the instrument (see 7.4),

*t* = time in seconds, and

*B* = viscometer coefficient (the kinetic energy correction).

**7.3.1** The coefficient of kinetic energy for the test viscometer shall be calculated as follows:

$$B = \frac{t_1 \times t_2}{t_2^2 - t_1^2} (V_2 t_1 - V_1 t_2)$$

where

*t*<sub>1</sub> = efflux time (200 seconds, *Min*) for an oil having a kinematic viscosity of *V*<sub>1</sub>, and

*t*<sub>2</sub> = efflux time for an oil having a kinematic viscosity of *V*<sub>2</sub> (about five times greater than *V*<sub>1</sub>).

NOTE — With viscometers of *C* constant greater than 0.05 and with a minimum efflux time of 200 seconds, *B/t* is not greater than 0.1 percent of the total, and may be neglected; then the equation of 7.3 becomes *V* = *Ct*.

### 7.4 Determination of the Viscometer Constant *C*

**7.4.1** Unless the constant is already known, calibrate the viscometer using primary standard, namely oil samples the viscosities of which have been determined by the National Physical Laboratory, New Delhi, or any other institution recognized by the Government of India.

**7.4.2** Determine, in the viscometer being calibrated, the efflux time of the appropriate standard, which shall not be less than 200 seconds by the method as prescribed in 7.2. Then calculate the constant  $C$  as follows by substituting the efflux time  $t$  and the kinematic viscosity of the oil:

$$\text{Viscometer constant, } C = \frac{V + B/t}{t}$$

where

$V$  = kinematic viscosity of the calibrating oil in centistokes,

$B$  = viscometer coefficient ( the kinematic energy correction ),  
and

$t$  = efflux time in seconds.

NOTE — With viscometer of  $C$  constant greater than 0.05 and with a minimum efflux time of 200 seconds,  $B/t$  is not greater than 0.1 percent of the total and may be neglected, then the equation in 7.4.2 becomes  $C = V/t$ .

## 8. REFRACTIVE INDEX

**8.1 Outline of the Method** — Refractive index is determined with an Abbe or Butyro refractometer.

### 8.1 Apparatus

**8.1.1 Abbe or Butyro Refractometer** — The temperature of the refractometer shall be controlled to within  $\pm 0.1^\circ\text{C}$  and for this purpose it shall be provided with a thermostatically controlled water-bath and a motor-driven pump to circulate water through the instrument. The instrument shall be standardized, following the manufacturer's instructions, with a liquid of known purity and refractive index or with a glass prism of known refractive index. Distilled water, which has a refractive index of 1.333 0 at  $20.0^\circ\text{C}$ , is a satisfactory liquid for standardization.

**8.1.2 Light Source** — If the refractometer is equipped with a compensator, a tungsten lamp or a daylight bulb shall be used. Otherwise, a monochromatic light, such as sodium vapour lamp shall be used.

**8.2 Procedure** — Filter the material through a filter paper to remove any impurities and the last traces of moisture. Make sure that the sample is completely dry. Adjust the temperature of the refractometer to  $40.0^\circ \pm 0.1^\circ\text{C}$  or to any other desired temperature. Ensure that the prisms are clean and completely dry, and then place a few drops of the material on the lower prism. Close the prisms, tighten firmly with the screw-head and allow to stand for 1 or 2 minutes. Adjust the instrument and light to obtain the most distinct reading possible and determine the refractive index.

**8.3 Temperature Corrections** — Unless the correction factors are specified in the detailed specification, approximate corrections shall be made using the following equation :

$$R = R' + K(T' - T)$$



where

$R$  = the reading of the refractometer reduced to the specified temperature,  $T^{\circ}\text{C}$ ;

$R'$  = the reading at  $T'^{\circ}\text{C}$ ;

$T'$  = the temperature at which the reading  $R'$  is taken;

$T$  = the specified temperature (generally  $40.0^{\circ}\text{C}$ ); and

$K$  =  $0.000\ 385$  (if Abbe refractometer is used); or  
 =  $0.58$  (if Butyro refractometer is used).

#### 8.4 Conversion of Butyro Refractometer Readings to Refractive Indices —

When Butyro refractometer is used, its readings ( $B$ ) shall be converted into refractive indices ( $n_D$ ) using Table 3.

### 9. ACID VALUE

**9.0 Outline of the Method** — Acid value is determined by finding the number of milligrams of potassium hydroxide required to neutralize free fatty acids contained in 1 g of the material.

#### 9.1 Reagents

**9.1.1 Ethanol-benzene Mixture** — Mix equal volumes of ethanol (95 percent) and benzene and neutralize this mixture with ethanolic potassium hydroxide solution ( $0.1\ \text{N}$ ) in the presence of phenolphthalein indicator.

**9.1.2 Standard Alcoholic Potassium Hydroxide Solution** —  $0.01\ \text{N}$ .

**9.1.3 Phenolphthalein Indicator Solution** — Dissolve  $0.1\ \text{g}$  of phenolphthalein in  $100\ \text{ml}$  of 60 percent rectified spirit (conforming to IS : 323-1959\*).

**9.2 Procedure** — Weigh accurately, to the nearest  $0.01\ \text{g}$ , about  $10\ \text{g}$ , depending upon the free acids, of the material into a conical flask. Add  $150\ \text{ml}$  of ethanol-benzene mixture to the material, shake and warm, if necessary. Titrate the solution with standard alcoholic potassium hydroxide solution using phenolphthalein indicator.

#### 9.3 Calculation

$$\text{Acid value} = 56.1 \frac{VN}{M}$$

where

$V$  = volume in ml of standard alcoholic potassium hydroxide solution used,

$N$  = normality of standard potassium hydroxide solution, and

$M$  = mass in g of the material taken for the test.

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\*Specification for rectified spirit (*revised*).

**TABLE 3 CONVERSION OF BUTYRO REFRACTOMETER READINGS  
(B) TO REFRACTIVE INDICES (nD)**

( Clause 8.4 )

B	nD	B	nD	B	nD	B	nD	B	nD
0.0	1.422 0	5.5	1.426 4	11.0	1.430 8	16.7	1.435 2	22.5	1.439 6
0.1	1.422 1	5.6	1.426 5	11.1	1.430 9	16.8	1.435 3	22.6	1.439 7
0.2	1.422 2	5.7	1.426 6	11.3	1.431 0	17.0	1.435 4	22.7	1.439 8
0.4	1.422 3	5.9	1.426 7	11.4	1.431 1	17.1	1.435 5	22.9	1.439 9
0.5	1.422 4	6.0	1.426 8	11.5	1.431 2	17.2	1.435 6	23.0	1.440 0
0.6	1.422 5	6.1	1.426 9	11.6	1.431 3	17.4	1.435 7	23.2	1.440 1
0.7	1.422 6	6.2	1.427 0	11.8	1.431 4	17.5	1.435 8	23.3	1.440 2
0.9	1.422 7	6.4	1.427 1	11.9	1.431 5	17.6	1.435 9	23.4	1.440 3
1.0	1.422 8	6.5	1.427 2	12.0	1.431 6	17.8	1.436 0	23.5	1.440 4
1.1	1.422 9	6.6	1.427 3	12.2	1.431 7	17.9	1.436 1	23.7	1.440 5
1.2	1.423 0	6.8	1.427 4	12.3	1.431 8	18.0	1.436 2	23.8	1.440 6
1.4	1.423 1	6.9	1.427 5	12.4	1.431 9	18.2	1.436 3	23.9	1.440 7
1.5	1.423 2	7.0	1.427 6	12.5	1.432 0	18.3	1.436 4	24.1	1.440 8
1.6	1.423 3	7.1	1.427 7	12.7	1.432 1	18.4	1.436 5	24.2	1.440 9
1.7	1.423 4	7.2	1.427 8	12.8	1.432 2	18.5	1.436 6	24.3	1.441 0
1.9	1.423 5	7.4	1.427 9	12.9	1.432 3	18.7	1.436 7	24.5	1.441 1
2.0	1.423 6	7.5	1.428 0	13.0	1.432 4	18.8	1.436 8	24.6	1.441 2
2.1	1.423 7	7.6	1.428 1	13.2	1.432 5	18.9	1.436 9	24.7	1.441 3
2.2	1.423 8	7.7	1.428 2	13.3	1.432 6	19.1	1.437 0	24.8	1.441 4
2.4	1.423 9	7.9	1.428 3	13.5	1.432 7	19.2	1.437 1	25.0	1.441 5
2.5	1.424 0	8.0	1.428 4	13.6	1.432 8	19.3	1.437 2	25.1	1.441 6
2.6	1.424 1	8.1	1.428 5	13.7	1.432 9	19.5	1.437 3	25.2	1.441 7
2.7	1.424 2	8.2	1.428 6	13.8	1.433 0	19.6	1.437 4	25.4	1.441 8
2.8	1.424 3	8.4	1.428 7	14.0	1.433 1	19.7	1.437 5	25.5	1.441 9
3.0	1.424 4	8.5	1.428 8	14.1	1.433 2	19.8	1.437 6	25.6	1.442 0
3.1	1.424 5	8.6	1.428 9	14.2	1.433 3	20.0	1.437 7	25.8	1.442 1
3.2	1.424 6	8.7	1.429 0	14.4	1.433 4	20.1	1.437 8	25.9	1.442 2
3.3	1.424 7	8.9	1.429 1	14.5	1.433 5	20.3	1.437 9	26.1	1.442 3
3.5	1.424 8	9.0	1.429 2	14.6	1.433 6	20.4	1.438 0	26.2	1.442 4
3.6	1.424 9	9.1	1.429 3	14.7	1.433 7	20.5	1.438 1	26.3	1.442 5
3.7	1.425 0	9.2	1.429 4	14.9	1.433 8	20.6	1.438 2	26.5	1.442 6
3.8	1.425 1	9.4	1.429 5	15.0	1.433 9	20.8	1.438 3	26.6	1.442 7
4.0	1.425 2	9.5	1.429 6	15.1	1.434 0	20.9	1.438 4	26.7	1.442 8
4.1	1.425 3	9.6	1.429 7	15.3	1.434 1	21.1	1.438 5	26.9	1.442 9
4.2	1.425 4	9.8	1.429 8	15.4	1.434 2	21.2	1.438 6	27.0	1.443 0
4.3	1.425 5	9.9	1.429 9	15.5	1.434 3	21.3	1.438 7	27.1	1.443 1
4.5	1.425 6	10.0	1.430 0	15.6	1.434 4	21.4	1.438 8	27.3	1.443 2
4.6	1.425 7	10.1	1.430 1	15.8	1.434 5	21.6	1.438 9	27.4	1.443 3
4.7	1.425 8	10.3	1.430 2	15.9	1.434 6	21.7	1.439 0	27.5	1.443 4
4.8	1.425 9	10.4	1.430 3	16.0	1.434 7	21.8	1.439 1	27.7	1.443 5
5.0	1.426 0	10.5	1.430 4	16.2	1.434 8	22.0	1.439 2	27.8	1.443 6
5.1	1.426 1	10.6	1.430 5	16.3	1.434 9	22.1	1.439 3	27.9	1.443 7
5.2	1.426 2	10.7	1.430 6	16.4	1.435 0	22.2	1.439 4	28.1	1.443 8
5.4	1.426 3	10.9	1.430 7	16.6	1.435 1	22.4	1.439 5	28.2	1.443 9

( Continued )

**TABLE 3 CONVERSION OF BUTYRO REFRACTOMETER READINGS  
(B) TO REFRACTIVE INDICES (nD)—Contd**

B	nD	B	nD	B	nD	B	nD	B	nD
28.3	1.444 0	34.4	1.448 4	40.6	1.452 8	46.9	1.457 2	53.4	1.461 6
28.5	1.444 1	34.6	1.448 5	40.7	1.452 9	47.0	1.457 3	53.6	1.461 7
28.6	1.444 2	34.7	1.448 6	40.9	1.453 0	47.2	1.457 4	53.7	1.461 8
28.7	1.444 3	34.9	1.448 7	41.0	1.452 1	47.3	1.457 5	53.9	1.461 9
28.9	1.444 4	35.0	1.448 8	41.1	1.453 2	47.5	1.457 6	54.0	1.462 0
29.0	1.444 5	35.1	1.448 9	41.3	1.453 3	47.6	1.457 7	54.2	1.462 1
29.2	1.444 6	35.3	1.449 0	41.4	1.453 4	47.7	1.457 8	54.3	1.462 2
29.3	1.444 7	35.5	1.449 1	41.5	1.453 5	47.9	1.457 9	54.5	1.462 3
29.4	1.444 8	35.6	1.449 2	41.7	1.453 6	48.0	1.458 0	54.6	1.462 4
29.6	1.444 9	35.7	1.449 3	41.8	1.453 7	48.2	1.458 1	54.8	1.462 5
29.7	1.445 0	35.8	1.449 4	42.0	1.453 8	48.3	1.458 2	55.0	1.462 6
29.9	1.445 1	36.0	1.449 5	42.1	1.453 9	48.5	1.458 3	55.1	1.462 7
30.0	1.445 2	36.1	1.449 6	42.3	1.454 0	48.6	1.458 4	55.3	1.462 8
30.1	1.445 3	36.3	1.449 7	42.4	1.454 1	48.8	1.458 5	55.4	1.462 9
30.3	1.445 4	36.4	1.449 8	42.5	1.454 2	48.9	1.458 6	55.6	1.463 0
30.4	1.445 5	36.5	1.449 9	42.7	1.454 3	49.1	1.458 7	55.7	1.463 1
30.6	1.445 6	36.7	1.450 0	42.8	1.454 4	49.2	1.458 8	55.9	1.463 2
30.7	1.445 7	36.8	1.450 1	43.0	1.454 5	49.4	1.458 9	56.0	1.463 3
30.8	1.445 8	37.0	1.450 2	43.1	1.454 6	49.5	1.459 0	56.2	1.463 4
30.9	1.445 9	37.1	1.450 3	43.3	1.454 7	49.7	1.459 1	56.3	1.463 5
31.0	1.446 0	37.2	1.450 4	43.4	1.454 8	49.8	1.459 2	56.5	1.463 6
31.2	1.446 1	37.4	1.450 5	43.6	1.454 9	50.0	1.459 3	56.6	1.463 7
31.4	1.446 2	37.5	1.450 6	43.7	1.455 0	50.1	1.459 4	56.8	1.463 8
31.5	1.446 3	37.7	1.450 7	43.9	1.455 1	50.2	1.459 5	56.9	1.463 9
31.6	1.446 4	37.8	1.450 8	44.0	1.455 2	50.4	1.459 6	57.1	1.464 0
31.8	1.446 5	37.9	1.450 9	44.2	1.455 3	50.5	1.459 7	57.3	1.464 1
31.9	1.446 6	38.1	1.451 0	44.3	1.455 4	50.7	1.459 8	57.4	1.464 2
32.1	1.446 7	38.2	1.451 1	44.4	1.455 5	50.8	1.459 9	57.6	1.464 3
32.2	1.446 8	38.3	1.451 2	44.6	1.455 6	51.0	1.460 0	57.7	1.464 4
32.3	1.446 9	38.5	1.451 3	44.7	1.455 7	51.1	1.460 1	57.9	1.464 5
32.5	1.447 0	38.6	1.451 4	44.9	1.455 8	51.3	1.460 2	58.0	1.464 6
32.6	1.447 1	38.7	1.451 5	45.0	1.455 9	51.4	1.460 3	58.2	1.464 7
32.8	1.447 2	38.9	1.451 6	45.2	1.456 0	51.6	1.460 4	58.3	1.464 8
32.9	1.447 3	39.0	1.451 7	45.3	1.456 1	51.7	1.460 5	58.5	1.464 9
33.0	1.447 4	39.2	1.451 8	45.5	1.456 2	51.9	1.460 6	58.6	1.465 0
32.2	1.447 5	39.3	1.451 9	45.6	1.456 3	52.0	1.460 7	58.8	1.465 1
33.3	1.447 6	39.5	1.452 0	45.7	1.456 4	52.2	1.460 8	58.9	1.465 2
33.5	1.447 7	39.6	1.452 1	45.9	1.456 5	52.3	1.460 9	59.1	1.465 3
33.6	1.447 8	39.7	1.452 2	46.0	1.456 6	52.5	1.461 0	59.2	1.465 4
33.7	1.447 9	39.9	1.452 3	46.2	1.456 7	52.7	1.461 1	59.4	1.465 5
33.9	1.448 0	40.0	1.452 4	46.3	1.456 8	52.8	1.461 2	59.5	1.465 6
34.0	1.448 1	40.1	1.452 5	46.4	1.456 9	53.0	1.461 3	59.7	1.465 7
34.2	1.448 2	40.3	1.452 6	46.6	1.457 0	53.1	1.461 4	59.8	1.465 8
34.3	1.448 3	40.4	1.452 7	46.7	1.457 1	53.3	1.461 5	60.0	1.465 9

(Continued)

TABLE 3 CONVERSION OF BUTYRO REFRACTOMETER READINGS

(B) TO REFRACTIVE INDICES (nD) — *Contd*

B	nD	B	nD	B	nD	B	nD	B	nD
60.2	1.466 0	67.5	1.470 7	75.0	1.475 5	83.1	1.480 1	91.4	1.484 8
60.3	1.466 1	67.7	1.470 8	75.1	1.475 5	83.2	1.480 2	91.6	1.484 9
60.5	1.466 2	67.8	1.470 9	75.3	1.475 6	83.4	1.480 3	91.8	1.485 0
60.6	1.466 3	68.0	1.471 0	75.5	1.475 7	83.6	1.480 4	92.0	1.485 1
60.8	1.466 4	68.1	1.471 1	75.6	1.475 8	83.8	1.480 5	92.1	1.485 2
60.9	1.466 5	68.3	1.471 2	75.8	1.475 9	83.9	1.480 6	92.3	1.485 3
61.1	1.466 6	68.4	1.471 3	76.0	1.476 0	84.1	1.480 7	92.5	1.485 4
61.2	1.466 7	68.6	1.471 4	76.1	1.476 1	84.3	1.480 8	92.7	1.485 5
61.4	1.466 8	68.7	1.471 5	76.3	1.476 2	84.3	1.480 9	92.9	1.485 6
61.5	1.466 9	68.9	1.471 6	76.3	1.476 3	84.6	1.481 0	93.0	1.485 7
61.7	1.467 0	69.1	1.471 7	76.7	1.476 4	84.8	1.481 1	93.2	1.485 8
61.8	1.467 1	69.2	1.471 8	76.8	1.476 5	85.0	1.481 2	93.4	1.485 9
62.0	1.467 2	69.4	1.471 9	77.0	1.476 6	85.2	1.481 3	93.6	1.486 0
62.2	1.467 3	69.5	1.472 0	77.2	1.476 7	85.3	1.481 4	93.8	1.486 1
62.4	1.467 4	69.7	1.472 1	77.3	1.476 8	85.5	1.481 5	94.0	1.486 2
62.5	1.467 5	69.9	1.472 2	77.5	1.476 9	85.7	1.481 6	94.1	1.486 3
62.6	1.467 6	70.0	1.472 3	77.7	1.477 0	85.9	1.481 7	94.3	1.486 4
62.8	1.467 7	70.2	1.472 4	77.9	1.477 1	86.0	1.481 8	94.5	1.486 5
62.9	1.467 8	70.3	1.472 5	78.1	1.477 2	86.2	1.481 9	94.7	1.486 6
63.1	1.467 9	70.5	1.472 6	78.2	1.477 3	86.4	1.482 0	94.8	1.486 7
63.2	1.468 0	70.7	1.472 7	78.4	1.477 4	86.6	1.482 1	95.0	1.486 8
63.4	1.468 1	70.8	1.472 8	78.6	1.477 5	86.7	1.482 2	95.2	1.486 9
63.5	1.468 2	71.0	1.472 9	78.7	1.477 6	86.9	1.482 3	95.4	1.487 0
63.7	1.468 3	71.1	1.473 0	78.9	1.477 7	87.1	1.482 4	95.6	1.487 1
63.8	1.468 4	71.3	1.473 1	79.1	1.477 8	87.3	1.482 5	95.8	1.487 2
64.0	1.468 5	71.4	1.473 2	79.2	1.477 9	87.5	1.482 6	96.0	1.487 3
64.2	1.468 6	71.6	1.473 3	79.4	1.478 0	87.6	1.482 7	96.1	1.487 4
64.3	1.468 7	71.8	1.473 4	79.5	1.478 1	87.8	1.482 8	96.3	1.487 5
64.5	1.468 8	71.9	1.473 5	79.8	1.478 2	88.0	1.482 9	96.5	1.487 6
64.7	1.468 9	72.1	1.473 6	80.0	1.478 3	88.2	1.483 0	96.7	1.487 7
64.8	1.469 0	72.2	1.473 7	80.1	1.478 4	88.3	1.483 1	96.9	1.487 8
65.0	1.469 1	72.4	1.473 8	80.3	1.478 5	88.5	1.483 2	97.0	1.487 9
65.1	1.469 2	72.5	1.473 9	80.5	1.478 6	88.7	1.483 3	97.2	1.488 0
65.3	1.469 3	72.7	1.474 0	80.6	1.478 7	88.9	1.483 4	97.4	1.488 1
65.4	1.469 4	72.9	1.474 1	80.8	1.478 8	89.1	1.483 5	97.6	1.488 2
65.6	1.469 5	73.0	1.474 2	81.0	1.478 9	89.2	1.483 6	97.8	1.488 3
65.7	1.469 6	73.2	1.474 3	81.2	1.479 0	89.4	1.483 7	98.0	1.488 4
65.9	1.469 7	73.3	1.474 4	81.3	1.479 1	89.6	1.483 8	98.1	1.488 5
66.1	1.469 8	73.5	1.474 5	81.5	1.479 2	89.8	1.483 9	98.3	1.488 6
66.2	1.469 9	73.7	1.474 6	81.7	1.479 3	90.0	1.484 0	98.5	1.488 7
66.4	1.470 0	73.8	1.474 7	81.9	1.479 4	90.2	1.484 1	98.7	1.488 8
66.5	1.470 1	74.0	1.474 8	82.0	1.479 5	90.3	1.484 2	98.9	1.488 9
66.7	1.470 2	74.1	1.474 9	82.2	1.479 6	90.5	1.484 3	99.1	1.489 0
66.8	1.470 3	74.3	1.475 0	82.4	1.479 7	90.7	1.484 4	99.2	1.489 1
67.0	1.470 4	74.5	1.475 1	82.5	1.479 8	90.9	1.484 5	99.4	1.489 2
67.2	1.470 5	74.6	1.475 2	82.7	1.479 9	91.1	1.484 6	99.6	1.489 3
67.3	1.470 6	74.8	1.475 3	82.9	1.480 0	91.2	1.484 7	99.8	1.489 4
								100.0	1.489 5

## 10. SAPONIFICATION VALUE

**10.1 Outline of the Method** — Saponification value is determined by finding the number of milligrams of potassium hydroxide required for the saponification of 1 g of the material.

### 10.1 Reagents

**10.1.1 Standard Alcoholic Potassium Hydroxide Solution** — 0.5 N.

**10.1.2 Standard Hydrochloric Acid** — 0.5 N.

**10.1.3 Phenolphthalein Indicator Solution** — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent of rectified spirit (conforming to IS : 323-1959\*).

**10.2 Procedure** — Weigh accurately, to nearest 0.001 g, about 2 g of the material into a conical flask. Add 25 ml of standard alcoholic potassium hydroxide solution to the flask by means of a burette or any other suitable instrument of equal precision. Connect the flask with a condenser, reflux for 1 hour, shaking from time to time, and wash the condenser with neutral alcohol. Titrate the solution, while still warm, with standard hydrochloric acid solution in the presence of phenolphthalein as indicator. Carry out a blank by refluxing and titrating exactly 25 ml of alcoholic potassium hydroxide solution under the same conditions.

### 10.3 Calculation

$$\text{Saponification value} = 56.1 \frac{(V_2 - V_1) N}{M}$$

where

$V_2$  = volume in ml of standard hydrochloric acid required for the titration of the blank,

$V_1$  = volume in ml of standard hydrochloric acid required for the titration of the material,

$N$  = normality of standard hydrochloric acid, and

$M$  = mass in g of the material taken for the test.

## 11. IODINE VALUE

**11.0 Outline of the Method** — The iodine value of an oil is a measure of its unsaturation. It is determined by the absorption of iodine under specified conditions and is expressed in grams of iodine absorbed per 100 g of the material. For oils without conjugated double bonds, Wijs method for deter-

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\*Specification for rectified spirit (revised).

mination of unsaturation (iodine value) is most commonly used. For oils with conjugated double bonds, for example, tung oil, the Wijs method is not reliable, as the values obtained are low. Therefore, in case of oils with conjugated double bonds the Woburn method is prescribed in 11.2 shall be followed.

## 11.1 Wijs Method

### 11.1.1 Reagents

11.1.1.1 *Potassium dichromate* — conforming to IS : 250-1964\*.

11.1.1.2 *Concentrated hydrochloric acid* — conforming to IS : 265-1976†.

11.1.1.3 *Potassium iodide solution* — Prepare a fresh solution for dissolving 10 g of potassium iodide free from potassium iodate, in 90 ml of water.

11.1.1.4 *Starch solution* — Triturate 5 g of soluble starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 litre of boiling water. Boil for 3 minutes. Allow to cool and decant off the supernatant clear liquid.

11.1.1.5 *Standard sodium thiosulphate solution* — approximately 0.1 N. Dissolve approximately 24.8 g of sodium thiosulphate crystals ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) in water which has been well boiled to free it from carbon dioxide and make up to 1 000 ml. Store the solution in a cool place in a dark-coloured stock bottle with a guard-tube filled with soda lime. After storing the solution for about 2 weeks, filter, if necessary, and standardize it as prescribed below :

Weigh accurately about 5 g of finely ground potassium dichromate which has been previously dried to a constant mass at  $105 \pm 2^\circ\text{C}$  into a clean 1-litre volumetric flask. Dissolve in water, make up to the mark; shake thoroughly and keep the solution in a cool dark place. For standardization of sodium thiosulphate, pipette 25 ml of this solution into a clean glass-stoppered 250-ml conical flask or bottle. Add 5 ml of concentrated hydrochloric acid and 15 ml of 10 percent potassium iodide solution. Allow to stand in the dark for 5 minutes and titrate the mixture with the solution of sodium thiosulphate, using starch solution as an internal indicator towards the end. The end point is taken when the blue colour changes to green. Calculate the normality (N) of the sodium thiosulphate solution as follows :

$$N = \frac{25 N}{49.03 V}$$

\*Specification for potassium bichromate, technical and analytical reagent (*revised*).

†Specification for hydrochloric acid (*second revision*).

where

$M$  = mass in g of the potassium dichromate, and

$V$  = volume in ml of sodium thiosulphate solution required for the titration.

#### 11.1.1.6 Iodine crystals — re-sublimed.

**11.1.1.7 Acetic acid** — glacial, 99 percent, having a melting point of  $14.8^{\circ}\text{C}$  and free from reducing impurities. Determine the melting point of the acetic acid and test it for reducing impurities as follows :

- a) *Determination of melting point* — Take a 15 cm long test tube and fill it to about two-thirds with the acetic acid. Insert into the acid a thermometer through a cork stopper fitting the test tube. The amount of acid shall be at least double the quantity required to cover the bulb of the thermometer when the bottom of the latter is 12 mm from the bottom of the test tube. Cool the acid by immersing the assembly in ice water until the temperature is  $10^{\circ}\text{C}$ , then withdraw the assembly from the ice water and stir the acid rather vigorously for a few moments, thus causing the super-cooled liquid to crystallize partially. Take thermometer readings every 15 seconds and take the temperature at which the reading remains constant for at least 2 minutes.

NOTE — An engraved stem thermometer calibrated between  $10^{\circ}\text{C}$  and  $65^{\circ}\text{C}$  and in  $0.1^{\circ}\text{C}$  intervals and with the  $0^{\circ}\text{C}$  point marked on the stem, is recommended. The thermometer shall have an auxiliary reservoir at the upper end, a length of about 370 mm and a diameter of about 6 mm.

- b) *Test for reducing impurities (potassium permanganate test)* — Dilute 2 ml of the acetic acid with 10 ml of water, add 0.1 millilitre of 0.1N potassium permanganate solution and maintain at  $27 \pm 2^{\circ}\text{C}$ . The material shall be taken as free from reducing impurities if the pink colour is not discharged at the end of 2 hours.

#### 11.1.1.8 Chlorine gas — dry.

#### 11.1.1.9 Iodine trichloride ( $\text{ICI}_3$ )

#### 11.1.1.10 Iodine monochloride ( $\text{ICI}$ ) — 98 percent.

**11.1.1.11 Wijs iodine monochloride solution** — Prepare this solution by one of the following three methods and store in a glass-stoppered bottle in a cool place, protected from light:

- a) Dissolve 13 g of iodine in 1 litre of acetic acid, using gentle heat, if necessary, and determine the strength by titration with standard sodium thiosulphate solution. Set aside 50 to 100 ml of the solution and introduce chlorine gas into the remainder until the characteristic colour change occurs and the halogen content is nearly doubled as ascertained again by titration. If the halogen content

has been more than doubled, reduce it by adding the requisite quantity of the iodine-acetic acid solution. A slight excess of iodine does no harm, but avoid an excess of chlorine.

Example:

If the titration of 20 ml of original iodine-acetic acid solution requires 22 ml of standard sodium thiosulphate, 20 ml of the finished Wijs solution should require between 43 and 44 ml (and not more than 44 ml) of the same sodium thiosulphate solution.

- b) Dissolve 8 g of iodine trichloride in approximately 450 ml of acetic acid. Dissolve separately 9 g of iodine in 450 ml of acetic acid, using heat, if necessary. Add gradually the iodine solution to the iodine trichloride until the colour has changed to reddish-brown. Add 50 ml more of iodine solution and dilute the mixture with acetic acid till 10 ml of the mixture is equivalent to 20 ml of standard thiosulphate solution when the halogen content is estimated by titration in the presence of an excess of potassium iodide and water. Heat the solution to 100°C for 20 minutes and cool. Prevent access of water vapour in preparing the solution.
- c) Dissolve 10 ml of iodine monochloride in about 1 800 ml of glacial acetic acid and shake vigorously. Using safety pipette, take 5 ml of this, add 10 ml of potassium iodide solution and titrate with 0.1 N standard sodium thiosulphate solution, using starch solution as indicator. Adjust the volume of the solution till it is approximately 0.2 N.

#### 11.1.1.12 Carbon tetrachloride or chloroform — Insert to Wijs solution.

**11.1.2 Procedure** — Filter the material through a filter paper to remove any impurities and the last traces of moisture. Make sure that the material as well as the glass apparatus used is absolutely clean and dry. Weigh accurately, by difference, an appropriate quantity of the oil between the limits indicated in col 2 and 3 of Table 4, into a clean dry 500-ml iodine flask or ground-glass stoppered bottle to which 25 ml of carbon tetrachloride or chloroform have been added and agitate to dissolve the contents. The mass of the material shall be such that there is an excess of 50 to 60 percent of Wijs solution over that actually needed. Add 25 ml of the Wijs solution and replace the glass stopper after wetting with potassium iodide solution; swirl for intimate mixing, and allow to stand in the dark for 1 hour. Carry out a blank test simultaneously under similar experimental conditions. After standing add 15 ml of potassium iodide solution and 100 ml of water, rinsing in the stopper also and titrate the liberated iodine with standard sodium thiosulphate solution, swirling the contents of the bottle continuously to avoid any local excess until the colour of the solution is straw yellow.



Add 1 ml of the starch solution and continue the titration until the blue colour formed disappears after thorough shaking with the stopper on.

**TABLE 4 MASS OF DRYING OIL TO BE TAKEN FOR DETERMINATION OF IODINE VALUE**

( Clause 11.1.2 )

IODINE VALUE EXPECTED	MASS OF MATERIAL		WEIGHING ACCURACY
	Maximum	Minimum	
(1)	(2)	(3)	(4)
	g	g	g
Less than 3	10.0	10.0	±0.001
5	6.346 0	5.077 0	±0.000 5
10	3.173 0	2.538 4	±0.000 2
50	0.661 2	0.528 8	±0.000 2
100	0.317 3	0.253 8	±0.000 1
150	0.212 5	0.170 0	±0.000 1
200	0.158 6	0.126 0	±0.000 1

## 11.2 Woburn Method

### 11.2.1 Reagents

#### 11.2.1.1 Chloroform

#### 11.2.1.2 Iodine crystals — re-sublimed.

#### 11.2.1.3 Bromine solution

#### 11.2.1.4 Potassium iodide solution — same as in 11.1.1.3.

#### 11.2.1.5 Starch solution — same as in 11.1.1.4.

#### 11.2.1.6 Standard sodium thiosulphate solution — same as in 11.1.1.5.

#### 11.2.1.7 Glacial acetic acid — same as in 11.1.1.7.

**11.2.1.8 Woburn solution (iodine bromide in glacial acetic acid) —** Dissolve 25.4 g of iodine in 900 ml of glacial acetic acid either by agitation in a volumetric flask or preferably in a conical flask with the aid of magnetic stirrer, and with gentle heating, if necessary. Tare at the same time a flask containing 50 ml of glacial acetic acid, and weigh 16.0 g of bromine into this flask. Mix the bromine solution with the iodine solution and make the whole up to 1 litre with glacial acetic acid.

**11.2.2 Procedure —** Weigh the material accurately, to the nearest 0.000 1 g, into a dry conical flask. The mass of the material to be taken shall depend on the iodine value of the material and shall be in accordance with

Table 4 given under 11.1.2. However, in the case of tung oil take about 0.06 to 0.08 g of the material. Dissolve this material in 10 ml of chloroform and keep the flask moving for about 5 minutes in an ice-water bath. While keeping the flask moving in the bath, add exactly 25 ml of the Woburn solution by means of a burette. It is absolutely essential to cool the flask and its contents to 0°C before adding Woburn solution. Close the flask with a ground-glass stopper, while keeping the flask in the bath. To prevent loss of halogen by evaporation, moisten the glass stopper with a drop of potassium iodide solution, taking care that no trace of this enters the reaction mixture. Keep the flask moving for 3 minutes in the ice-water bath. Fill the bath with crushed ice, so that the flask is packed in ice just below its neck. Keep the whole in a dark place for three hours. After this period, add 10 ml of potassium iodide solution and 50 ml of water and swirl. Titrate the contents of the flask with standard sodium thiosulphate solution until the yellow colour has almost disappeared, add 1 ml of the starch solution and continue the titration until the blue colour has just disappeared. Carry out a blank under the same conditions.

### 11.3 Calculation

$$\text{Iodine value} = \frac{12.69 (V_2 - V_1) N}{M}$$

where

$V_2$  = volume in ml of standard sodium thiosulphate solution required for the blank,

$V_1$  = volume in ml of standard sodium thiosulphate solution required for the material,

$N$  = normality of the standard sodium thiosulphate solution, and

$M$  = mass in g of the material taken for the test.

## 12. UNSAPONIFIABLE MATTER

**12.1 Outline of the Method** — The material is completely saponified with alcoholic potassium hydroxide solution and extracted with petroleum hydrocarbon solvent. The petroleum hydrocarbon solvent extract is washed with aqueous alcohol and water. The washed ether extract is evaporated and the residue weighed. Unsaponifiable matter is this residue minus the fatty acids present in it, which are determined by titration with sodium hydroxide solution in alcoholic medium. By this method only unsaponifiable substances not volatile at 100°C are determined. In view of the possible presence of metallic soaps in the oil, for example, in boiled linseed oil, the method includes a washing with nitric acid. However, in the absence of metallic soaps, the washings with nitric acid may be omitted.

## 12.1 Apparatus

**12.1.1 Flat-bottomed or Conical Flask** — 250 or 300 ml capacity. An ordinary round, flat-bottomed flask, fitted with a long glass tube which acts as a condenser, may also be used.

### 12.1.2 Reflux Condenser

**12.1.3 Separating Funnels** — 500 ml capacity.

## 12.2 Reagents

**12.2.1 Alcoholic Potassium Hydroxide Solution** — Dissolve 70 to 80 g of potassium hydroxide in an equal quantity of water and add sufficient aldehyde-free ethyl alcohol (95 per cent  $v/v$ ) or aldehyde-free rectified spirit (conforming to IS : 323-1959\*), prepared as described under G-3.4 of IS : 323-1959\*, to make up to 1 000 ml. Allow to stand overnight, decant the clear liquid and keep in a bottle closed tightly with a cork or rubber stopper.

**12.2.2 Ethyl Alcohol** — 95 percent ( $v/v$ ), or rectified spirit (conforming to IS : 323-1959\*).

**12.2.3 Phenolphthalein Indicator Solution** — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit (conforming to IS : 323-1959\*).

**12.2.4 Petroleum Hydrocarbon Solvent** — conforming to solvent grade, 60/80 of IS : 1945-1978†.

**12.2.5 Aqueous Alcohol** — containing 10 percent ( $v/v$ ) of ethyl alcohol (conforming to IS : 321-1964‡).

**12.2.6 Dilute Nitric Acid** — 1 : 3 ( $v/v$ ).

**12.2.7 Standard Sodium Hydroxide Solution** — approximately 0.02 N.

**12.2.8 Acetone** — free from evaporation residue (see IS : 170-1976§).

**12.3 Procedure** — Weigh accurately, to the nearest 0.1 g, in a flask, about 5 g of the well-mixed sample, previously warmed and filtered, if necessary. Add 50 ml of alcoholic potassium hydroxide solution. Connect the flask with a reflux condenser and reflux the mixture gently for 1 hour or until the saponification is complete, shaking from time to time. Wash the condenser with about 10 ml of ethyl alcohol. Remove the condenser from the flask and transfer the contents of the flask to a separating funnel. Rinse the flask several times with a total of 50 ml of water and pour the water into the

\*Specification for rectified spirit (*revised*).

†Specification for petroleum hydrocarbon solvents (*second revision*).

‡Specification for ethyl alcohol (absolute alcohol) (*revised*).

§Specification for acetone (*second revision*).

separating funnel. Rinse the flask several times with a total of 50 ml of petroleum hydrocarbon solvent and pour the petroleum hydrocarbon solvent also into a separating funnel. Allow the material in the separating funnel to cool to room temperature, stopper the funnel, shake vigorously for 1 minute and allow to stand until the two layers have clearly separated. Transfer the lower layer to another separating funnel and repeat the extraction with petroleum hydrocarbon solvent solution at least six times more using 50 ml of petroleum hydrocarbon solvent for each extraction. If any emulsion is formed, add a small quantity of ethyl alcohol or alcoholic potassium hydroxide solution.

**12.3.1** Collect all the petroleum hydrocarbon extracts in a separating funnel. Wash the combined extracts in a funnel three times with 25 ml portions of aqueous alcohol shaking vigorously and drawing off the alcohol-water layer after each washing. Wash with 25 ml of dilute nitric acid, if necessary. Again wash ether layer successively with 25 ml portions of water until the wash-water no longer turns pink on addition of two drops of phenolphthalein indicator solution. Transfer petroleum hydrocarbon layer to a tared flask containing few pieces of pumice stone, and evaporate to dryness on a water-bath. To remove the last traces of moisture, add a few millilitres of acetone and pass a gentle stream of clean dry air over the surface of the material or evacuate using a water vacuum pump at 50°C for about 15 minutes. Cool in a desiccator and weigh. Repeat the evacuating, cooling and weighing until the difference between the two weighings is less than 0.5 mg.

**12.3.2** Dissolve the residue in 50 ml of warm natural ethyl alcohol and titrate the solution with standard sodium hydroxide solution in the presence of phenolphthalein as indicator.

## 12.4 Calculation

**12.4.1** Mass in g of the fatty acids in the extract =  $B = 0.282 V N$

where

$V$  = volume in ml of standard sodium hydroxide solution used,  
and

$N$  = normality of standard sodium hydroxide solution.

**12.4.2** Unsaponifiable matter,  
percent by mass =  $\frac{100 (A - B)}{M}$

where

$A$  = mass in g of the residue (see 12.3.1)

$B$  = mass in g of the fatty acids in the extracts (see 12.4.1), and

$M$  = mass in g of the material taken for the test.

### 13. ACETYL VALUE

**13.0 Outline of the Method** — The material is acetylated by refluxing with acetic anhydride and the excess anhydride is decomposed with water and sodium bicarbonate solution. The saponification value of the washed and dried acetylated oil is determined as in 10.

#### 13.1 Apparatus

**13.1.1 Beaker** — 800 ml capacity.

**13.1.2 Separating Funnel** — 50 ml capacity.

**13.1.3 Conical Flasks** — 250 to 300 ml capacity.

**13.1.4 Reflux Condenser** — Any efficient reflux condenser, at least 65 cm long.

**13.1.5 Sand-bath or Electric Hot-plate with Thermostatic Control**

#### 13.2 Reagents

**13.2.1 Acetic Anhydride** — containing 95 to 100 percent (by mass) of acetic anhydride. Determine the acetic anhydride content as given in 13.2.1.1 and 13.2.1.2.

**13.2.1.1** Weigh accurately about 2 g of the acetic anhydride into a 200-ml glass-stoppered conical flask, cool in ice and add 5 ml of freshly distilled aniline. Insert the glass stopper immediately, shake vigorously and allow to stand at room temperature for 30 minutes. Wash down the sides of the flask with 50 ml of ice-cold water. Mix well and titrate with previously standardized 1 *N* sodium hydroxide solution using phenolphthalein as indicator until the pink colour persists for 10 minutes.

$$A = \frac{\text{Volume of ml of 1 } N \text{ sodium hydroxide solution required}}{\text{Mass in g of acetic anhydride taken for the test}}$$

**13.2.1.2** Weigh accurately about 2 g of the acetic anhydride into another flask, add 50 ml of water, allow to stand for 30 minutes and titrate with the standard sodium hydroxide solution to the same end point as above, using phenolphthalein as indicator.

$$B = \frac{\text{Volume in ml of 1 } N \text{ sodium hydroxide solution required}}{\text{mass in g of acetic anhydride taken for the test}}$$

$$\begin{aligned} &\text{Acetic anhydride} \\ &\text{percent by mass} = 10 \cdot 209 (B - A) \end{aligned}$$

#### 13.2.2 Carbon Dioxide or Nitrogen Gas

**13.2.3 Sodium Bicarbonate Solution** — freshly prepared 0.5 percent (*m/v*) and neutral to litmus.

**13.2.4 Anhydrous Sodium Sulphate****13.2.5 Standard Alcoholic Potassium Hydroxide Solution — 0.5 N.**

**13.3. Procedure** — Weigh accurately about 10 g of the material in a conical flask, add 20 ml of acetic anhydride and boil the mixture under a reflux air condenser for about 2 hours on the sand-bath or electric hot-plate. Pour the mixture into a beaker containing 500 ml of water and boil for 15 minutes. Bubble a stream of carbon dioxide or nitrogen through the mixture during boiling to prevent bumping. Discontinue boiling, cool slightly, and remove the water with a siphon. Add another 500 ml of water and boil again. Discontinue boiling, cool and transfer the contents of the beaker to a separating funnel and reject the lower layer. Wash the acetylated sample successively (a) three times with 50 ml of water, (b) twice with 50 ml of sodium bicarbonate solution, and (c) twice with 50 ml of warm water (60 to 70°C). Drain and remove as much of the water as possible, and then transfer the acetylated sample to a beaker and add approximately 5 g of anhydrous sodium sulphate. Allow to stand for about one hour with occasional stirring. Filter through a dry filter paper, preferably in an oven at 100 to 110°C, remove the filter paper and keep the sample in the oven until it is thoroughly dry. Determine the saponification values of the original material and the acetylated product by the procedure prescribed in 10.2.

$$\text{Acetyl value} = \frac{S' - S}{1.00000 - 0.00075 S}$$

where

$S'$  = saponification value after acetylation, and

$S$  = saponification value before acetylation.

**14. FREEDOM FROM MINERAL ACID**

**14.1 Outline of the Method** — Freedom from mineral acid is determined by adding methyl orange indicator to the water extract of the material.

**14.1 Reagents**

**14.1.1 Methyl Orange Indicator** — Dissolve 0.01 g in 100 ml of water (see IS : 2263-1962\*).

**14.2 Procedure** — Shake thoroughly about 100 g of the material with 100 ml of hot water (90 to 100°C) in a separating funnel. Separate and draw off the aqueous layer. Test the aqueous layer with methyl orange indicator. The material shall be taken to be free from mineral acids, if the aqueous layer does not give red colour with methyl orange indicator.

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\*Methods of preparation of indicator solutions for volumetric analysis.

## 15. DRYING TIME

**15.0 Outline of the Method** — The material shall be tested for surface drying after the panel, which has been brushed with the material, is exposed to the deffused daylight for a specified period.

### 15.1 Apparatus

**15.1.1 Ground-glass Panels** — Unless specified otherwise in the material specification, glass panels shall be  $105 \times 150$  mm or  $150 \times 75$  mm in size. They shall be reasonably plain and free from surface irregularities. They shall be cleaned in the manner prescribed in 15.1.1.1 before use.

**15.1.1.1** All oil or grease shall be removed from the panel by dipping in benzene and rubbing with a soft and clean rag. The panel shall be rinsed thoroughly with water and well washed with soap and water, until the wetted panel shows no water breaks when held in a vertical position. The panel shall then be rinsed with alcohol and allowed to dry in the air in a dust-free chamber.

**15.2 Procedure** — Brush the oil on the ground surface of the ground-glass panel. Place the panel in the vertical position in a well-ventilated room so that its surface is illuminated by diffused daylight for at least 6 hours during each day of drying period. Carry out the test at room temperature under such conditions that at any time during testing the relative humidity of the room does not exceed 65 percent. Examine the panel for surface drying after specified interval as specified in the material specification ( *see* 2.5 ).

## 16. HEAT TEST

**16.1 Outline of the Method** — Heat test is applied to find gel time of the drying oils and it is the time required for the oil to form a solid gel under specified conditions of temperature.

### 16.1 Apparatus

**16.1.1 Test Tubes** —  $150 \times 16$  mm, weighing  $15 \pm 2$  g, with a mark near the bottom to indicate 5 ml level and each tube closed by a perforated cork so that a glass rod, 3 mm in diameter, shall move through it freely.

**16.1.2 Thermometer** — short stem, half opal, nitrogen filled, satisfying the following requirements:

Overall length	$210 \pm 10$ mm
Scale length	$110 \pm 10$ mm
Bulb length	5 to 10 mm
Stem diameter	5 to 6 mm
Bulb diameter	5 to 5.5 mm

The scale to read from 245 to 305°C graduated to 0.2°C.

The graduations to permit an immersion of 80 mm.

**16.1.3 Oil-Bath** — consisting of a tall type 1000-ml beaker ( *see* IS : 2619-1971\* ) with a suitable cover plate having three holes, two for the test tubes and one for the thermometer. The thermometer may be suspended in the bath by a wire or may be sealed to a glass rod. No stirrer is used in the bath. A screen around the bath enables the temperature to be reached more readily. Fill the beaker with 700 ml of cottonseed oil ( *see* IS : 543-1968† ) or any other suitable oil. When the bath oil has become tarry and viscid after use, it shall be renewed; otherwise heating may be irregular.

**16.1.4 Glass Rods** — 3 mm in diameter.

**16.2 Procedure** — Assemble the apparatus as shown in Fig. 7. Place the thermometer so that the bulb is in level with the bottom of the test tubes. Heat the bath to a temperature of 293°C, rapidly at the start and slowly when it approaches this temperature. Place the two tubes, each containing 5 ml of the material, into the large hole of the cover and note the time. Remove the source of heat for about 45 seconds and then re-apply. Before 2 minutes have elapsed the temperature of the bath will have fallen to 282°C at which point it should be kept as steady as possible. When the samples have been in the bath for 9 minutes, raise the glass rods at intervals of 45 seconds. Note the time when each sample becomes firmly set. At this point the oil shall be so stiff that the entire tube may be lifted by the aid of the rod. As setting or jellying takes place within a few seconds of fluidity, a good end point is afforded. Note the time when the material has completely set.

**16.2.1** Use the material obtained from this test for further test for extractable matter under 17.

**16.3 Report** — Note the time when the material has completely set and report whether the gel is sticky or dry.

## 17. EXTRACTABLE MATTER

**17.0 Outline of the Method** — Extractable matter is determined by evaporating the petroleum hydrocarbon solvent extract of the gelled material.

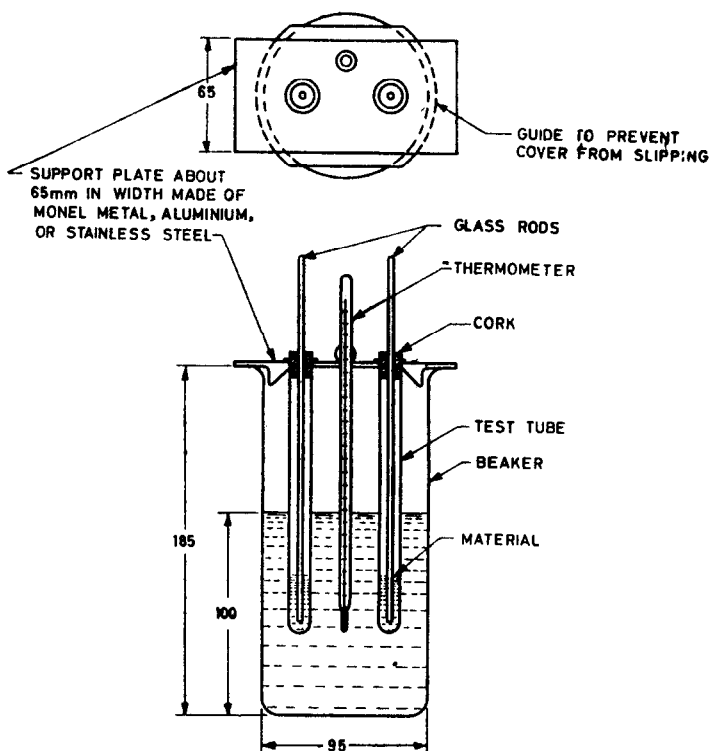
### 17.1 Reagents

**17.1.1 Petroleum Hydrocarbon Solvent** — conforming to solvent grade 60/80 of IS : 1745-1978\*.

\*Specification for glass beaker ( *first revision* ).

†Specification for cottonseed oil ( *second revision* ).





All dimensions in millimetres

FIG. 7 ASSEMBLY OF APPARATUS FOR HEAT TEST

**17.2 Procedure** — Raise the temperature of the bath at the conclusion of the test prescribed in 16.2 to 290°C and leave the tube and its contents undisturbed in the oil-bath for a further period of 20 minutes from the time when gelation is observed. Withdraw the tube containing the sample from the bath and clamp it in a vertical position to cool, with the cork removed. On cooling the gel generally detaches itself from the walls of the tube and can be withdrawn with the rod; otherwise break the tube away from the gel. Cut off and discard the oxidized top surface, and crumble and mix the remainder. Accurately weigh about 2 g into a mortar, grind as finely as possible with a little sand, previously washed and ignited till well mixed, and transfer to a Soxhlet extractor thimble, using a little petroleum hydrocarbon solvent, if necessary. Extract the gel for not less than an hour with petroleum hydrocarbon solvent. Transfer the petroleum hydrocarbon solvent extract completely to a tared dish containing pieces of pumice stones and evaporate

to dryness on a water-bath. To remove the last traces of petroleum hydrocarbon solvent, place the dish in an air oven at 80 to 90°C for about 1 hour. Cool in a desiccator and weigh. Repeat drying and cooling until the difference between the two weighings is less than 0.5 mg.

### 17.3 Calculation

$$\begin{array}{l} \text{Extractable matter,} \\ \text{percent by mass} = 100 \times \frac{m}{M} \end{array}$$

where

$m$  = mass in g of residue obtained after evaporating the petroleum hydrocarbon solvent extract, and

$M$  = mass in g of the material taken for the test.

## 18. INSOLUBLE BROMIDE TEST

**18.1 Outline of the Method** — This test is carried by adding bromine solution to the material maintained at low temperature.

### 18.1 Reagents

**18.1.1 Petroleum Hydrocarbon Solvent** — conforming to solvent grade 60/80 of IS : 1745-1978\*.

#### 18.1.2 Bromine

**18.2 Procedure** — Dissolve 1 ml of clear oil in 20 ml of petroleum hydrocarbon solvent in a test tube or a small flask. Mix the solution thoroughly and place the container in water at 0°C in case of tung oil, and in freezing mixture with a temperature of -10°C in case of linseed oil. Add bromine drop by drop from a burette avoiding vigorous action until a considerable excess is present, as indicated by the persistence of a strong red colour. Shake well the container and then allow to stand in cold water for 15 minutes.

**18.2.1** The material shall be taken to have passed this test if the solution remains perfectly clear.

## 19. ASH CONTENT

**19.1 Outline of the Method** — Ash content is determined by igniting the oil, under specified conditions and weighing the non-combustible material.

**19.1 Procedure** — Weigh a clean and previously ignited porcelain crucible or dish, accurately to the nearest 0.0001 g. Add 10 to 25 ml of the material and weigh again. Heat the material gently for bringing a flame on the bottom and sides of the crucible until the oil ignites. Reduce the size of the flame

\*Specification for petroleum hydrocarbon solvents (second revision).

until the heat is just sufficient to keep the material burning. Continue heating of the sample to a black char and transfer it to a muffle furnace or heat it over a burner until all carbonaceous matter is reduced to ash. Remove the crucible, place in a desiccator and cool to room temperature. Weigh and repeat heating and cooling until the difference between two weighings is less than 1 mg.

## 19.2 Calculation

$$\text{Ash content, percent by mass} = 100 \frac{m}{M}$$

where

$m$  = mass in g of the ash content, and

$M$  = mass in g of the material taken for the test.

## 20. FOOTS

**20.1 Outline of the Method** — The material to be tested is treated under specified conditions with an acid-calcium chloride solution and acetone. This mixture is allowed to settle in a graduated tube and the foots content is determined volumetrically. The test for foots shall be applied to (a) oils as received, (b) heated oils, and (c) chilled oils.

### 20.1 Apparatus

**20.1.1 Graduated Tube** — having an internal diameter of 10 to 15 mm and a capacity of not less than 70 ml. The graduation in 0.1 ml shall extend at least from 10 to 50 ml above the bottom of the tube.

### 20.2 Reagents

**20.2.1 Acetone** — conforming to IS : 170-1976\*.

**20.2.2 Acid-calcium Chloride Solution** — Dissolve 600 g of calcium chloride as  $\text{CaCl}_2$  or 800 g as  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  in a mixture of 80 ml of concentrated hydrochloric acid (conforming to IS : 265-1976†) and 500 ml of water. Dilute to 1 litre and mix. Filter, if the solution is not clear.

### 20.3 Procedure

**20.3.1 Oils as Received** — Cool all the materials to a temperature between 20 to 27°C. Shake thoroughly the bottle containing the material and transfer 25 ml of the material to the graduated tube. Add 25 ml of acetone and 10 ml of acid-calcium chloride solution and shake exactly for 1 minute.

\*Specification for acetone (second revision).

†Specification for hydrochloric acid (second revision).

Clamp the tube in a vertical position and allow the solution to settle for 24 hours at a temperature between 20 to 27°C. Determine the volume of stratum line between clear calcium chloride solution and the clear acetone and oil mixture to the nearest of 0.1 ml or fraction thereof.

**20.3.2 Heated Oils** — Heat a portion of the material to  $65 \pm 2^\circ\text{C}$  for 10 minutes. Cool to room temperature and determine the foots by the method prescribed in 20.3.1.

**20.3.3 Chilled Oils** — Heat a portion of the material to  $65 \pm 8^\circ\text{C}$  for 10 minutes. Transfer it to a clean and dry bottle. Stopper tightly and keep it in ice or water mixture at  $0^\circ\text{C}$  for exactly two hours. Then place it in a constant temperature bath at  $25.0 \pm 0.5^\circ\text{C}$  for 30 minutes. Determine the foots by the method prescribed in 20.3.1.

**20.4 Calculation** — Multiply the volume of foots by four to obtain the amount of foots present as a percentage by volume.

## 21. FREEDOM FROM BREAK

**21.0 Outline of the Method** — Freedom from break is determined by primary and secondary break test by noting the mucilaginous precipitate formation when oil is heated to  $280^\circ\text{C}$  and cooled with or without the addition of a few drops of glycerine. Break test in drying oils ensures that the portion of oil is rendered insoluble when the oil is heated to polymerization or varnish-making temperature.

### 21.1 Procedure

**21.1.1 Primary Break Tests** — Take 15 ml of the material in a 100-ml heat resistant breaker (see IS : 2619-1971\*) and suspend a thermometer in the centre of the beaker so that the bulb is completely immersed in the material and does not touch the sides of the beaker. Heat the beaker to  $280^\circ\text{C}$  over an open flame in such a way that the temperature is attained within 10 minutes. After heating to  $280^\circ\text{C}$ , withdraw the flame. Cool to room temperature.

**21.1.2 Secondary Break Test** — If there is no break in the primary break test, the experiment is repeated with the oil after adding 2 to 3 drops of glycerine.

**21.1.3** The material shall be considered free from break if no mucilaginous precipitate appears in the material on cooling.

## 22. FREEDOM FROM LEAD

**22.0 Outline of the Method** — Determination of lead is carried out by treating the ash of the material with nitric acid and then with ammonium acetate solution and precipitating lead as lead sulphide and finally oxidizing to lead sulphate.

\*Specification for glass beakers (first revision).

## 22.1 Reagents

**22.1.1 Dilute Nitric Acid** — lead-free, of strength approximately 4 N.

**22.1.2 Ammonium Acetate Solution** — lead-free, 10 percent ( $m/v$ ).

**22.1.3 Concentrated Sulphuric Acid** — lead-free, relative density 1.84, conforming to IS : 266-1977\*.

**22.1.4 Dilute Sulphuric Acid** — 2 percent obtained by dissolving 11 ml of concentrated sulphuric acid in 1 litre of water.

**22.1.5 Ammonium Acetate** — solid, lead-free.

**22.1.6 Hydrogen Sulphide** — gas.

**22.1.7 Hydrogen Sulphide Water** — saturated solution.

**22.2 Procedure** — Weigh 50 g of the material in silica basin and ash it carefully until only slight traces of carbon remain. The temperature of the basin shall not be allowed to rise above faint red heat, as at higher temperatures some lead may be lost by volatilization. Treat the ash so obtained with dilute nitric acid. The quantity of acid is immaterial provided it is sufficient to extract the soluble matter, but avoid too great an excess since it has to be evaporated off. Allow the basin to stand on a boiling water-bath for at least 3 hours. In case a large quantity of insoluble residue is left, heat the basin on the water-bath overnight. Decant off the supernatant liquid through a filter paper and extract the insoluble residue again on a boiling water-bath for one hour with dilute nitric acid. Filter through the same filter paper and wash the residue thoroughly on the filter paper with hot water. Treat the residue on the filter paper with 10 ml of ammonium acetate solution, filter and wash again. Mix the filtrate and washings in a 500-ml evaporating basin, add 2 ml of concentrated sulphuric acid and evaporate the contents of the basin on a sand bath till fumes appear. Add 100 ml of water to the basin and allow to stand on the boiling water for 15 minutes. Then dilute the contents to about 150 ml and allow to stand overnight at room temperature. Filter the insoluble matter on a No. 42 Whatman filter paper (9 cm) and wash thoroughly with dilute sulphuric acid. Transfer the filter paper and residue to a small beaker, cover with 20 ml of water, and add 1 to 2 g of ammonium acetate. Heat the beaker on the water-bath for not less than half an hour, stirring the contents occasionally. Decant the liquid through a No. 42 Whatman filter paper (9 cm). Repeat the extraction with water and ammonium acetate. Transfer all the insoluble matter including the filter pulp to the filter paper and wash thoroughly with warm water collecting the filtrate and washings in a 150-ml beaker. Pass hydrogen sulphide through the liquid for 10 to 15 minutes and filter the precipitated lead sulphide at once through a No. 40 Whatman filter paper (9 cm). Wash thoroughly but quickly with hydrogen sulphide water keeping the residue on the filter paper,

\*Specification for sulphuric acid (second revision).

if any, covered with liquid till washing is completed. Transfer the precipitate and filter paper to a tared silica crucible. Dry carefully, ignite to sulphate, cool and weigh.

**22.3** Alternatively, determine the quantity of lead by either of the following methods :

- a) By electrolysis after dissolving the precipitated lead sulphide in nitric acid, or
- b) By polarographic method.

**22.4 Calculation** — Calculate as lead (Pb) and express the result as percentage of the mass of the material taken for the test.

## **23. FREEDOM FROM ROSIN ( COLOPHONY )**

**23.0 Outline of the Method** — Freedom from rosin ( colophony ) is determined by treating the material with acetic anhydride and a few drops of dilute sulphuric acid and noting the change in colour.

### **23.1 Reagents**

**23.1.1 Acetic Anhydride**

**23.1.2 Dilute Sulphuric Acid** — 1 : 1 ( v/v ).

**23.2 Procedure** — Take about 5 ml of the material in a test tube and add an equal volume of acetic anhydride. Cool and add to it from a side of the test tube a few drops of dilute sulphuric acid, very slowly, taking care not to disturb the solution.

**23.2.1** The material shall be taken to have passed the test if no fugitive violet coloration appears.

## **24. TEST FOR COLOUR CHANGE ON HEATING**

**24.0 Outline of the Method** — Sample under test and approved sample are heated to a specified temperature and change in colour is determined using Lovibond tintometer.

### **24.1 Apparatus**

**24.1.1 Lovibond Tintometer**

**24.1.2 Thermometer** — 0 to 360°C.

**24.2 Procedure** — Take about 60 to 70 ml of sample in a 100-ml heat-resistant beaker. Suspend a clean thermometer in the oil and heat on a wire gauze preferably using 2 burners. Raise the temperature to 300°C in about

8 to 10 minutes. Take care to avoid local charring due to flames impinging on the sides of the beaker by stirring with a glass rod. Remove the source of heat after the temperature has just reached 300°C. Cool to room temperature and observe the colour of the sample in a 1-in. cell using the Lovibond tintometer. Similarly treat an approved sample and compare the colour.

## 25. TEST FOR FREEDOM FROM FISH OIL AND LINSEED OILS

**25.1 Outline of the Method** — Addition of bromine to unsaturated fatty acids of fish oil in a petroleum hydrocarbon solvent gives a precipitate. The formation of a precipitate of hexabromide, when the oil is treated with bromine in chloroform and then with alcohol and ether, indicates the presence of linseed oil.

### 25.2 Reagents

**25.2.1 Petroleum Hydrocarbon Solvent** — 145/205 (low aromatic), conforming to IS : 1745-1978\*.

**25.2.2 Bromine**

**25.2.3 Chloroform**

**25.2.4 Rectified Spirit** — conforming to IS : 323-1959†.

**25.2.5 Ether** — conforming to solvent grade of IS : 336-1973‡.

### 25.3 Procedure

**25.3.1 Test for Freedom from Fish Oil** — Remove the moisture present, if any, by filtering through double layer filter paper. Dissolve 2 to 3 ml of the sample in 20 ml of petroleum hydrocarbon solvent in a test tube. Mix well and keep in a freezer maintained at  $10 \pm 1^\circ\text{C}$  for 5 minutes. Add bromine drop by drop, from a burette avoiding vigorous action until a considerable excess is present as indicated by the presence of a strong red colour. Shake well and allow to stand in cold water at a temperature of  $25^\circ\text{C}$  for 15 minutes. The material shall be taken to have passed this test if no cloudiness develops.

**25.3.1.1** If cloudiness or a precipitate appears due to bromination of the sample, proceed as follows:

- a) Saponify 2 to 3 ml of the sample in a 100-ml Erlenmeyer flask adding 3 ml of sodium hydroxide solution (50 percent) and 5 ml of ethanol. Place the mixture on a steam-bath and heat for about an hour under reflux. Remove the condenser and remove the ethanol

\*Specification for petroleum hydrocarbon solvents (second revision).

†Specification for rectified spirit (revised).

‡Specification for ether (second revision).

avoiding evaporation to dryness. Add 40 ml of hot water and boil for an hour to remove the traces of ethanol. Pour the mixture into a 250-ml separatory funnel, washing the flask with 20 ml portion of boiling water. Acidify the mixture with 2 ml of 9 N sulphuric acid. Allow to settle until the fatty acids form a clear layer and the mixture is quite cold. Draw off the acid layer as completely as possible and wash the oil layer with 20 ml portions of saturated sodium chloride solution. Reject the washings. Add 20 ml of diethyl ether and dissolve the fatty acids by gently swirling the separatory funnel.

- b) Transfer the solution in a test tube and cool to  $10 \pm 1^\circ\text{C}$  for 5 minutes. Add bromine drop by drop from a burette avoiding vigorous action until a considerable excess is present as indicated by the presence of a strong red colour. Shake well and allow to stand in cold water at a temperature of  $25^\circ\text{C}$  for 15 minutes. Centrifuge or filter and wash the precipitate with 5 ml portions of ethyl ether. Dry to remove the ether. Check the melting point of the precipitate.
- c) The material shall be taken to have passed this test if the precipitate melts completely at 180 to  $183^\circ\text{C}$ , leaving no residue or blackening of the material.

**25.3.2 Test for Freedom from Linseed Oil** — Pipette 1 ml of sample in a dry test tube which may be fitted with a ground-glass stopper. Add 5 ml of chloroform and about 1 ml of bromine dropwise till red in colour. Cool the test tube in ice water-bath. Add about 1.5 ml of rectified spirit in drops with shaking until the precipitate which is formed just dissolves and then add 10 ml of ether. Mix and place the tube in the ice-bath for 30 minutes. Appearance of a precipitate or cloudiness indicates the presence of linseed oil.

**25.3.2.1** The requirement of this test shall be taken to have been satisfied if no precipitate or cloudiness appears.

## 26. TEST FOR FREEDOM FROM CASTOR OIL

**26.0 General** — The turbidity test described in Method A may be used for the rapid detection of castor oil in other oils. Method A is sensitive to the extent of 1 percent of castor oil, but badly oxidized vegetable oils, *MAHUA* oil and some varieties of sesame oil may also respond. A positive response shall be confirmed by Method B which is based on thin-layer chromatography. This is specific for castor oil, and has a sensitivity of 1 percent.

**26.1 Method A** — When castor oil is present in other oils, treatment of a petroleum ether solution of the oil with sulphuric acid, in the presence of ammonium molybdate, causes a turbidity to appear.



### 26.1.1 Reagents

**26.1.1.1 Acidified Petroleum Ether** — Add 2 ml of concentrated hydrochloric acid to 100 ml of petroleum ether (boiling range 40 to 60°C or 60 to 80°C).

**26.1.1.2 Sulphuric acid-molybdate reagent** — In 100 ml of concentrated sulphuric acid dissolve 1.25 g of ammonium molybdate.

**26.1.2 Procedure** — In a clean, dry test tube take about 1 ml of the oil and add 10 ml of acidified petroleum ether. Shake vigorously for 2 minutes and add one drop of the molybdate reagent. Instantaneous development of white turbidity indicates the likely presence of castor oil.

**26.2 Method B** — A triglyceride constituent of castor oil, perhaps triricinolein, gives a spot with a characteristic  $R_f$  value by suitable thin-layer chromatography.

### 26.2.1 Apparatus

**26.2.1.1 Slides** — two clean microscope slides ( $7.6 \times 2.5$  cm). Glass plates of  $20 \times 5$  cm or  $20 \times 10$  cm may also be used instead of microscope slides.

**26.2.1.2 Developing tank** — a tall form beaker of at least 10 cm height.

**26.2.1.3 Visualization tank** — A dry beaker or tank saturated with iodine vapour by placing a few crystals at the bottom and leaving for an hour.

### 26.2.2 Materials

**26.2.2.1 Silica gel containing binder** — passing 75-micron IS sieve; this is a mixture of silica gel (85 g) and calcium sulphate (15 g) as a binder. The mixed material is available commercially.

**26.2.2.2 Developing solvent** — a mixture of petroleum ether (boiling range 40 to 60°C), diethyl ether and acetic acid in the proportion of 60 : 40 : 2 (v/v).

**26.2.3 Procedure** — Hold the two microscope slides together face to face and dip them in a slurry of silica gel (45 g) in chloroform-methanol mixture (80 ml + 20 ml). Withdraw the slides, separate them and allow to dry in air for 10 minutes. At one end of the coated slides, apply 5 ml (50 mg) of the oil sample as a one-percent solution in chloroform using a capillary (melting point) tube. Dry briefly, and place the slide in the beaker containing 3 ml of the developing solvent mixture. Cover with a watch glass and allow the solvent to travel up to within 1 cm of the top of the plate (about 6 minutes). Remove from the tank, dry in air and place in the visualization tank containing iodine vapour. Occurrence of a spot with an  $R_f$  value of about 0.25 shows the presence of castor oil. All other spots will be above this. A prepared sample of an oil containing one percent of

added castor oil may be run alongside for convenience in identifying the spot. The spot shall be noted in the visualization tank, since it fades on removing.

## 27. TEST FOR FREEDOM FROM COTTONSEED OIL

**27.1 General** — Development of red colour on heating the oil with a solution of sulphur in carbon disulphide indicates the presence of cottonseed oil. The test is also responded by hampseed oils, kapok oil, ambadi seed oil and oils and fats containing fatty acids of cyclopropanoid structure.

### 27.2 Reagents

**27.2.1 Sulphur Solution** — Prepare one percent ( $m/v$ ) solution of sulphur in carbon disulphide and add an equal volume of amyl alcohol.

**27.3 Procedure** — Take about 5 ml of the material and add an equal volume of sulphur solution. Mix thoroughly by shaking and heat gently on a water-bath (70 to 80°C) for a few minutes with occasional shaking until the carbon disulphide has boiled off and the sample stops foaming. Place the tube in an oil-bath maintained at 110 to 115°C and hold for 1 or 2 hours. Development of red colour at the end of this period indicates the presence of cottonseed oil.

**27.3.1 Sensitivity** — This test is generally sensitive to the extent of 0.5 percent of cottonseed oil in other oils.

## 28. DETERMINATION OF ROSIN ACIDS

**28.1 Outline of the Method** — A known quantity of the material is saponified with caustic alkali and hydrolysed with hydrochloric acid. The rosin acids and fatty acids thus obtained are extracted with benzene, washed and selective esterification of fatty acids is carried out in the presence of rosin acids.

### 28.2 Reagents

**28.2.1 Alcoholic Potassium Hydroxide Solution** — 10 percent.

**28.2.2 Hydrochloric Acid** — relative density 1.19.

**28.2.3 Benzene**

**28.2.4 Acetone**

**28.2.5 Esterification Solution** — Mix 500 ml of *n*-butanol, 500 ml of benzene, and 6 g of concentrated sulphuric acid. Place the mixture in a flask, attach a moisture trap and reflux for 30 minutes to form butyl-sulphuric acid. Cool and store in a glass-stoppered bottle.

**28.2.6 Alcoholic Potassium Hydroxide Solution** — 0.2 to 0.25 N. accurately standardized.

**28.2.7 Indicator** — 0.05 percent phenolphthalein or thymol blue indicator.

### 28.3 Procedure

**28.3.1** To the weighed sample in 500-ml conical flask add 50 ml of alcoholic potassium hydroxide solution, attach to a suitable condenser and reflux on hot-water bath for 1 hour. After refluxing, remove the flask from the hot water-bath and cool to room temperature under tap water. Add 100 ml of water, then add 20 to 25 ml of hydrochloric acid. The solution shall be distinctly acidic. Reflux for 5 minutes again and cool.

**28.3.2** Transfer the sample quantitatively to a 1 litre separatory funnel, wash the conical flask with 50 ml of water, 25 ml of benzene, 50 ml of water and 15 ml of acetone respectively and add to the funnel. Shake, allow the layers to separate and draw off the lower aqueous layer into a second 1 litre separatory funnel. Extract the aqueous layer with a second 50 ml portion of benzene and drain the aqueous layer into a third 1 litre funnel. Add the second benzene extract to the first. Repeat the extraction of the aqueous layer with successive 50-ml portions of benzene (total number not less than 3). Discard the aqueous layer. Combine the benzene extracts and wash with three 50-ml portion of water or until the wash water is free from acid. Transfer the washed benzene extract to a weighed 250-ml conical flask with the aid of 25 ml of benzene. Evaporate the benzene on the steam-bath. Add 5 g of anhydrous ethyl alcohol to remove any water present by azeotropic distillation, cool and weigh.

**28.3.3** Weigh a portion of the benzene extract (28.3.2) accurately into a 250-ml conical flask with ground joint according to the following:

<i>Rosin Acids</i> percent	<i>Sample Size</i> g
0 to 5	5 to 8
5 to 20	3 to 5
20 to 50	2 to 3
50 to 100	1 to 2

Using a burette or constant delivery pipette, accurately measure 50 ml of esterification solution into the flask and add a few boiling stones. Attach a moisture trap and condenser, place on a hot-plate, heat to boiling and reflux for 20 minutes. The boiling shall be vigorous at all times, so that the water which is formed separates rapidly. At the end of the heating period allow the flask and contents to cool somewhat. Remove the flask, cool immediately

to room temperature, and titrate with alcoholic potassium hydroxide solution. Make a blank titration on the same volume of esterification solution after refluxing it in the same manner.

NOTE — Light coloured samples may be titrated with phenolphthalein or thymol blue indicator. Dark samples and those containing small amounts of mineral acids or alkalis are preferably titrated potentiometrically.

## 28.4 Calculation

$$\text{Rosin acids ( as abietic acid ),} \\ \text{percent by mass} = \frac{(A - B) \times N \times 30.78}{M} - 0.3$$

where

$A$  = volume in ml of alcoholic potassium hydroxide solution required for the sample,

$B$  = volume in ml of alcoholic potassium hydroxide solution required for the blank,

$N$  = normality of alcoholic potassium hydroxide solution,

$M$  = mass in g of the material taken for the test, and

0.3 = experimentally determined constant to correct for unesterified fatty acids.

## 29. TEST FOR PRESENCE OF BLOWN OIL

**29.1 Outline of the Method** — Sample is mixed with light petroleum hydrocarbon solvent and examined.

### 29.2 Apparatus

**29.2.1 Measuring Cylinder** — 50 ml capacity fitted with ground-glass joint.

### 29.3 Reagents

**29.3.1 Petroleum Hydrocarbon Solvent** — boiling range 40 to 60°C conforming to IS : 1745-1978\*.

**29.4 Procedure** — Take 10 ml of the sample in a 50-ml measuring cylinder and mix it thoroughly with 30 ml of light petroleum hydrocarbon solvent. Observe the clarity of the solution once in the beginning and once after three days' storage at room temperature.

**29.5 Report** — The sample shall be declared to have passed the test if the solution in initial observation is clear and it does not show any deposits after storage.

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\*Specification for petroleum hydrocarbon solvents ( *second revision* ).

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